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AD-A237 798

ENTATION PAGE

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2. REPORT DATE: 11 Feb 91

3. REPORT TYPE AND DATES COVERED: 1 Aug 89 - 31 July 90. Technical Rep.

4. TITLE AND SUBTITLE: The Growth of Ultrathin Epitaxial Intermetallic Films

5. FUNDING NUMBERS: AFOSR 89-0494

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8. PERFORMING ORGANIZATION REPORT NUMBER: AEOSR-TR- 91 0575

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES):
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AFOSR/NE Bldg 410
Bolling AFB
Washington, DC 20332

10. SPONSORING/MONITORING AGENCY REPORT NUMBER: 2306/B1

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT: unlimited

12b. DISTRIBUTION CODE

13. ABSTRACT *(Maximum 200 words)*

The growth of FeAl and NiAl intermetallic compounds on III-V substrates has been studied using reflection high-energy electron diffraction (RHEED), transmission electron diffraction (TEM), and selected area electron channelling patterns (SAECP). Procedures for the growth of both of these intermetallics with layer-by-layer control were developed. The quality of the films on InP(100) substrates exceeds those grown on GaAs(100) substrates due to the lower lattice mismatch. The films are stable to at least 820K. TEM measurements indicate that the burgers vector of misfit dislocations is a complete a[100]. SACP, TEM, and TEM indicate that the film relaxation approximates the Matthews Blakeslee prediction. Even this was surprising in light of the layer by layer growth after relaxation since the type of dislocation that forms cannot glide to the interface to relieve the strain. Preliminary semiconductor – intermetallic – semiconductor structures have been grown. The quality of the interfaces is being assessed.

14. SUBJECT TERMS: FeAl, Epitaxial Intermetallic

15. NUMBER OF PAGES: 9

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
Unclassified	Unclassified	Unclassified	UL

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
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91-04435

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Contents

1 Objective

The goals of this project are to develop a new class of ultrathin intermetallic films that form stable, abrupt interfaces with III-V semiconductors. These films would have application to metal–semiconductor barriers and contacts, to high speed devices, and to integrated magnetic structures. The possibility of buried, epitaxial semiconductors was to be investigated. The fundamental growth processes and stability issues were to be studied prior to the study of devices. Important questions to be answered included:

1. What was the role of lattice mismatch? Did the pseudomorphic critical thickness follow the Matthews Blakeslee prediction?
2. Did chemistry affect the epitaxial quality – were their differences between FeAl and NiAl?
3. How did the growth parameters affect the interface and film quality?
4. Could a growth model be developed?
5. Could a semiconductor be grown on top of the an epitaxial metal?

2 Status of Current Work

The growth of FeAl and NiAl on both InP and GaAs substrates has been investigated by reflection high-energy electron diffraction (RHEED), transmission electron diffraction (TEM), and selected area electron channelling patterns (SAECP). The stability of the films has been studied, role of lattice mismatch has been examined, the dislocation Burgers vector determined, and a partial elucidation of the growth modes has been obtained.

2.1 Growth Modes

Thin Films of Fe_xAl_{1-x} were first grown by molecular beam epitaxy (MBE) on InP. On the InP substrates the intermetallic films have a lattice mismatch of only -0.9% , while on the GaAs substrates the mismatch is 2.9% . Before growth of the epitaxial intermetallic alloy on the InP, a 0.2 μm InAlAs buffer and a ten layer AlAs diffusion barrier were deposited. Initial films were grown at 200°C and were stable to at

least 600°C. Upon the initiation of growth, the (RHEED) intensity was observed to decrease slowly and then exhibit oscillations corresponding to layer by layer growth. There was a short incubation period that depended on the Fe and Al flux ratio. The diffraction pattern was sharp, indicating that the FeAl surface was nearly as smooth as the starting AlAs surface. Upon heating to 550°C, the diffracted beams became sharper yet; growth on the annealed surface gave intensity oscillations that were an order of magnitude stronger. The detailed work is described in Wowchak et al., J. Vac. Sci. Technol., **B7**, 733 (1989).

To explore the role of lattice mismatch and film stoichiometry on the epitaxial process Fe_xAl_{1-x} was deposited on GaAs(100) substrates and studied with similar techniques. The main differences were that (1) the initial misfit was larger so that the lattice relaxation could be measured vs time, as will be described below and (2) the layer by layer growth was weaker than in the (nearly) lattice matched case. The results are described in Kuznia et al., J. Elect. Mater., **19**, 561 (1990). Like the case of InP substrates, RHEED intensity oscillations were observed with the strongest ocurring when x=y. It was also found that the growth mode involved bilayer growth and that there was some sort of transition in the growth mechanism at incident fluxes corresponding to the growth of Fe_3Al. That sharp films were prepared even after annealing to 600°C was taken as evidence of the stability of the interface. This was confirmed with TEM, though more work needs to be done over a larger interface area. The probably magnetic phase and transition in growth mode occuring at Fe_3Al needs to be studied and its magnetic properties determined.

By contrast NiAl does not give as strong intensity oscillations nor as sharp a diffraction pattern. It appears that in this case the exact nucleation conditions are important and that the growth of bulk phases is likely when slight deviations from stoichiometry occur. Thus the FeAl appears to be more forgiving as an intermetallic contact to semiconductors.

2.2 Strain Relaxation

For ultrathin films suitable for optoelectronics one needs pseudomorphic layers free of dislocations. For majority carrier transport in metal base transistors or in resonant tunnelling structures one may only need to limit the density of dislocations. As devices become smaller, this requirement is of course relaxed. To understand the generation and multiplication of dislocations in these films the lattice parameter was measured during growth and the dislocations were studied by TEM and SAECP after growth.

The surprises were that (1) the lattice relaxation occurred less quickly than

predicted by the classical Matthews–Blakeslee (MB) theory and (2) the form of what was then thought to be a different relaxation mechanism approximated the MB result. The relaxation measurement is given in Keller et al., J. Elect. Mater., submitted (1990) and is shown in Fig. 1. The lattice parameter during growth was measured by RHEED and compared to calculation assuming known elastic moduli. The MB result is equivalent to assuming either force balance or an array of dislocations somehow forming at the interface. Two measurements are shown

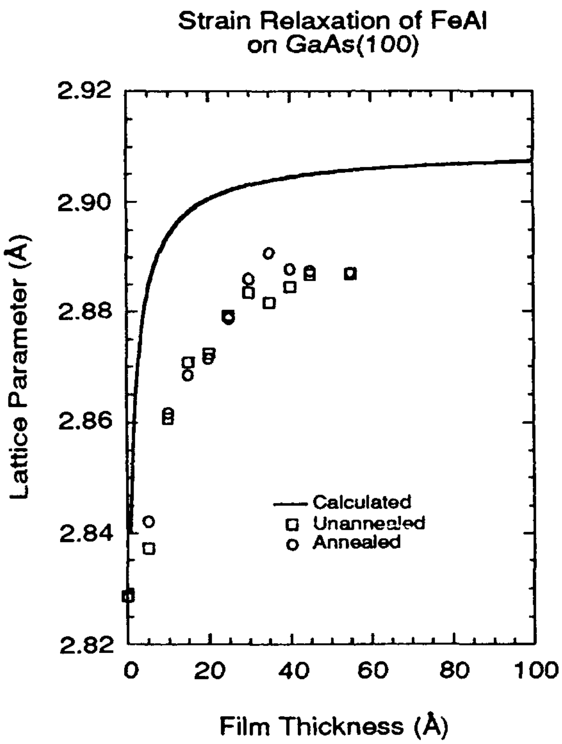

Figure 1: Lattice parameter of FeAl grown on GaAs(100) as a function of film thickness.

for each point. The unannealed point corresponds to the initial growth at a given thickness. The annealed is the lattice parameter measurement after heating to 550°C. The lack of further relaxation is taken as additional evidence of the stability of the films. This constitutes the first measurement of lattice relaxation in these materials.

To understand the detailed mechanism of the defect generation and to compare to the RHEED measurements of these slight changes in lattice parameter, TEM and SAECP measurements were made on the same films. The SAECP measurements in R.R. Keller et al., submitted to J. Elect. Mater., confirmed the RHEED measure-

ments. The TEM measurement in J.E. Angelo, et al., submitted to Appl. Phys. Lett., found that the Burgers vector of the dislocation was a[100]. Considering the slip system of the FeAl and GaAs this means that the dislocations were not due to threading dislocations from the substrate and that dislocations nucleated at the surface could not glide down to the interface to relieve the strain. Since the RHEED measurements indicate good layer by layer growth the dislocations could not come in at the edge of islands. Consequently we must conclude that the dislocations are"punched out" at the interface. This is an uncomfortable conclusion and we are investigating the depth dependence of the strain and reexamining the extent of the layer growth. The simplest way out of this difficulty is to postulate that the films grow by the formation of columns and that the dislocations come in at the base of the columns. Unfortunately, we have not seen any experimental evidence for this. This is a very important issue and is crucial to a number of current studies of the growth of strained films.

I summary, we know a great deal more about the growth of intermetallic films, including dislocation dynamics, dislocation types, growth mode. We do not yet have quantitative theories that explain the measurements. We are able to grow pseudomorphic films on InP substrates. We are able to grow films on GaAs substrates with fewer defects than expected. We have set up a strong program to understand the growth of this new class of materials.

2.3 Directions for Current Research

Our main effort now will be to try to understand the role of growth kinetics in growth of these intermetallic films. We will focus on

1. Migration enhanced epitaxy as a growth technique to promote defect free films
2. Ultrahigh vacuum metal organic chemical vapor deposition as a way to alter the growth kinetics: possibly achieving higher surface mobility of the reactants at low substrate temperatures
3. Patterned substrates for strain relief
4. Construction of a scanning tunnelling microscope to examine the real space, local structure of the FeAl surface
5. Investigation of the magnetic properties of the films and the transition from FeAl to Fe_3Al
6. Characterization of the structure of semiconductor–intermetallic–semiconductor sandwiches

3 Publications Resulting from this Program

3.1 Published, In press, and Submitted

1. A.M. Wowchak, J.N. Kuznia, and P.I. Cohen, "Layer by layer growth of FeAl on InP(100) Substrates," J. Vac. Sci. Technol., **B7**, 733 (1989)

2. J.N. Kuznia, A.M. Wowchak, and P.I. Cohen, "Epitaxy of FeAl Films on GaAs(100) by Molecular Beam Epitaxy," J. Elect. Mater. **19**, 561 (1990).

3. R.R. Keller, A.M. Wowchak, J.E. Angelo, J.N. Kuznia, P.I. Cohen, and W.W. Gerberich, "Growth and Characterization of Iron Aluminide Films on Compound Semiconductors," submitted to J. Elect. Mater., 1990.

4. J.E. Angelo, J.N. Kuznia, A.M. Wowchak, P.I. Cohen, and W.W. Gerberich, "The Structure and Identification of the Misfit Dislocations at an FeAl–AlAs–GaAs Interface Using Moiré-fringe Contrast in a TEM," Appl. Phys. Lett., (1991), in press.

5. J.E. Angelo, J.N Kuznia, A.M. Wowchak, P.I. Cohen, and W.W. Gerberich, "A Study of FeAl/AlAs/GaAs Interfaces using Moiré-Fringe Contrast in a Transmission Electron Microscope," Proc. Symp. Mater. Res. Soc., Boston, 1990, in press.

6. R.E. Keller, J.E. Angelo, A.M. Wowchak, P.I. Cohen, and W.W. Gerberich, "Electron Channelling Analysis of Strained Iron Aluminide Films," Proc. Symp. MRS Soc., Boston, 1990, in press.

3.2 Planned

1. N.K. Sapthotharan, H.D. He, and P.I. Cohen, "The growth of FeAl on GaAs by migration enhanced epitaxy,"

2. H.D. He, N.K. Sapthotharan, and P.I. Cohen, "Epitaxial growth of GaAs on FeAl,"

3. K.M. Chen and P.I. Cohen, "Ultrahigh vacuum chemical vapor growth of FeAl on GaAs using Alane,"

4 Personnel

Faculty

1. P.I. Cohen is the principal investigator leading the project
2. W.W. Gerberich in the Materials Science department at the University of Minnesota is expert at the role of defects in epitaxial films and is assisting us with defect characterization and modelling. No funds are being provided by the AFOSR.

Research Associates

1. H.D. He is currently leading the effort on the MBE growth of the intermetallics. His Ph.D. work was on the MBE growth of metal superlattices.
2. K.M. Chen is leading the effort on the growth using ultrahigh vacuum chemical vapor epitaxy. His Ph.D. work was on the MBE growth of Si.

Graduate Students

1. A.M. Wowchak developed the growth of the intermetallics and received a Ph.D. in Electrical Engineering, Dec., 1990. The title of his Ph.D. thesis was "The Growth of Ultrathin Intermetallic Films by MBE."
2. J.N. Kuznia developed the measurement of the strain relaxation and received an M.S. in Electrical Engineering, Dec., 1989. The title of his M.S. thesis was "The nucleation of Iron and Iron Aluminides on Compound Semiconductors."
3. N.K. Sapthotharan is a second year student working with H.D. He
4. Two new graduate students will replace Wowchak and Kuznia
5. J.E. Angelo is a graduate student in Materials Science doing the transmission electron microscopic characterization of the films. No funds are being provided by the AFOSR.
6. R.R. Keller is a graduate student in Materials Science characterizing the films by selected area electron channelling patterns. No funds are being provided by the AFOSR.

5 Presentations

1. J.N. Kuznia, A.M. Wowchak, and P.I. Cohen, "Epitaxy of FeAl Films on GaAs(100) by Molecular Beam Epitaxy," Elec. Mater. Conf., Boston, 1989.
2. W.-H. Liu, A.M. Wowchak, and P.I. Cohen, "Layer by layer Growth of NiAl on GaAs(100)," Electronic Materials Conference, Santa Barbara, 1990.
3. W.W. Gerberich and P.I. Cohen, (*invited*), "Growth and Characterization of Iron Aluminide films on Compound Semiconductors," Annual Meeting of the Minerals, Metals, and Materials Society, Anaheim, 1990.
4. J.E. Angelo, J.N. Kuznia, A.M. Wowchak, P.I. Cohen, and W.W. Gerberich, "A Study of FeAl/AlAs/GaAs Interfaces using Moiré-Fringe Contrast in a Transmission Electron Microscope," Symp. Mater. Res. Soc., Boston, 1990.
5. R.E. Keller, J.E. Angelo, A.M. Wowchak, P.I. Cohen, and W.W. Gerberich, "Electron Channelling Analysis of Strained Iron Aluminide Films," Symp. MRS Soc., Boston, 1990.
6. P.I. Cohen, (*invited*), to be presented at the Gordon Research Conference, "Dynamics of MBE Growth: *in situ* studies of the role of defects," New Hampshire, July, 1991.